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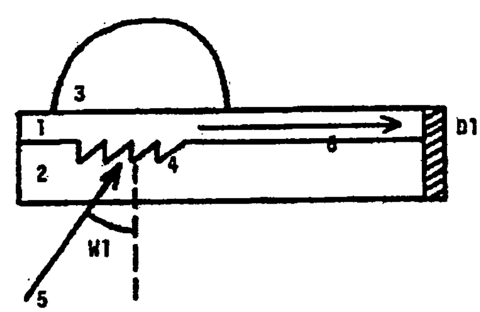
(54) Sensor for Detecting Changes in the Refractive Index of a Solid or Liquid Measurement Substance

(57) The sensor according to this invention for detecting changes in the refractive index of fluids and solids is constructed with integrated optical elements. As a waveguide structure, it consists of a waveguide film (1), a substrate (2) and a diffraction grating (4). A measurement substance (3) covers the waveguide film (1) at least in the area of the diffraction grating (4). The sensor principle is that a change in the refractive index of the measurement substance (3) produces a change in the effective refractive index N of a light wave (6), which is referred to as a mode, the light wave being guided in the waveguide film (1). To detect this change, the arrangement (1, 2, 3, 4) consisting of the waveguide structure and the measurement substance (3) is used as a grating input coupler and/or as a grating output coupler or as a Bragg reflector.



PATENT CLAIMS

1. A sensor for detecting changes in the real component and/or the imaginary component of the complex refractive index of a solid or liquid measurement substance, characterized in that it consists of a waveguide film (1), a substrate (2) and at least one diffraction grating (4) which functions as a grating coupler or a Bragg reflector; it is also constructed as a waveguide structure in the area of the diffraction grating (4), and it can be brought in contact with the measurement substance (3), at least in the area of the diffraction grating (4).

2. The sensor according to Claim 1, characterized in that the diffraction grating (4) is a phase volume grating.

3. The sensor according to Claim 1, characterized in that the diffraction grating (4) is a surface relief grating.

4. The sensor according to Claim 1, characterized in that the refractive index of the waveguide film (1) is selected to be at least 1%, but preferably more than 10%, higher than that of the substrate (2) in order to achieve a high sensitivity.

5. The sensor according to one of Claims 1 or 4, characterized in that the waveguide film (1) is covered with a protective layer (10) outside of the grating region to partially or completely prevent the mode from being influenced outside of the grating region.

6. Use of the sensor according to one of Claims 1 through 5 to detect changes in the real component and/or the imaginary component of the complex refractive index of a solid or liquid measurement substance, characterized in that the change in the effective refractive index of a mode (6) propagating in an arrangement consisting of the waveguide structure plus the measurement substance (3) is measured, said change being produced due to a change in the refractive index of the measurement substance (3).

7. Use according to Claim 6, characterized in that a laser beam (5) which is directed at the diffraction grating (4) at a fixed angle of incidence (W1) is input into the waveguide film (1) via this diffraction grating (4), and the changes in the effective refractive index produced by the changes in refractive index of the measurement substance (3) are determined by measuring the changes in the intensity of the mode (6).

8. Use according to Claim 6, characterized in that an angle of incidence (W1) at which a laser beam (5) is directed at the diffraction grating (4) is readjusted when there is a change in the refractive index of the measurement substance (3) so that the mode (6) has the greatest possible intensity and the change in the effective refractive index is determined from the change in the angle of incidence (W1).

9. Use according to Claim 6, characterized in that the mode (6) is output from the waveguide film (1) through the diffraction grating (4), and the change in the effective refractive index which occurs due to the change in the refractive index of the measurement substance (3) is determined by measuring the change in the output angle (W2) or, when the output angle (W2) is fixed, the change in the intensity of the output laser beam (7) is recorded.

10. Use according to Claim 6, characterized in that the mode (6) is directed at the diffraction grating (4) at a Bragg angle (W3) and is Bragg-reflected on the diffraction grating (4), and the change in the effective refractive index which is induced by the change in the refractive index of the measurement substance (3) is determined by measuring the change in intensity of the reflected mode (8) and/or the transmitted mode (9).

11. Use according to one of Claims 7 through 10, characterized in that the intensity of the mode (6) is determined by output of the mode via a second diffraction grating (13) and measuring the intensity of the laser beam (15) thus output by using a detector (D6).

DESCRIPTION

The present invention relates to a sensor according to the preamble of Patent Claim 1. A refractometer is a known device for detecting changes in the refractive index in liquids and solids by determining the total reflection angle between two media, with the reference medium consisting of a highly refractive prism having a known refractive index. This device takes up a relatively large amount of space and requires a relatively large measurement volume, which but this may be a great disadvantage when working with expensive measurement substances.

The present invention, as characterized in Patent Claim 1, achieves the object of creating a sensor which

1. can resolve changes in refractive index of liquids and solids up to an order of magnitude of 10^{-5};
2. needs only a very small measurement volume;
3. takes up the smallest possible amount of space.

This invention is explained in greater detail below on the basis of drawings as examples. They show:

Figure 1: a schematic diagram of the basic elements of this invention;

Figure 2: a measurement device according to this invention having a grating input coupler;

Figure 3: a measurement device according to this invention having a grating output coupler;

Figure 4: a measurement device according to this invention having a Bragg reflector;

Figure 5: a schematic diagram of the basic elements of this invention, where the waveguide is covered with a protective layer outside of the grating region;

Figure 6: a device according to this invention for measuring the intensity of the guided light wave, whereby the scattered light generated by the guided light wave is captured by fiber optics and sent to a detector;

Figure 7: a device according to this invention for measuring the intensity of the guided light wave, with the guided light wave being output via a second diffraction grating.

The basic component of the integrated optics is the planar waveguide which consists of a thin dielectric layer on a substrate. Input laser light can be guided in this thin layer by total reflection. The rate of propagation of such a guided light wave (referred to below as a "mode") amounts to c/N, where c is the velocity of light and N is the effective refractive index of the mode propagating in the waveguide. The effective refractive index N is determined first by the configuration of the waveguide (layer thickness and refractive index of the thin layer and refractive index of the substrate) and also by the refractive index of the measurement substance adjacent to the thin layer. The sensor principle is based on the fact that a change in configuration of the waveguide results in a change in the effective refractive index N. A change in the effective refractive index induced in this way can be detected for example with a grating input and/or a grating output or with a Bragg reflector. The mechanism of operation of the grating coupler and/or the Bragg reflector is described on the basis of the figures.

The basic elements of this invention are diagramed schematically in Figure 1. A thin layer is provided in the form of a planar waveguide film 1 (e.g., a glassy layer of SiO_2-TiO_2) on a substrate 2 (borosilicate glass, for example). Waveguide film 1 and substrate 2 together form waveguide 1/2. For laser light to propagate by total reflection in waveguide film 1, the refractive index of waveguide film 1 must exceed that of ambient media (i.e., substrate 2, measurement substance 3). The waveguide film 1 may have a microporous structure, obtainable by a dip coating process in film production, for example. A diffraction grating 4 of length L is provided on the surface of the waveguide film 1, facing either substrate 2 or measurement substance 3, or a diffraction grating may be provided in the volume of the waveguide film (for production of the waveguide and diffraction grating, see, for example, W. Lukosz and K. Tiefenthaler, *Optics Letter* 8 (1983), 537-539).

Diffraction grating 4 is used for diffraction of laser light, the diffraction being influenced significantly by the effective refractive index N.

Substance 3 which is to be tested, also called the "measurement substance," is deposited on waveguide film 1, at least in the region of the grating.

According to Figure 2, a laser beam 5 can be input into a waveguide 1/2 through a diffraction grating 4 and travel along the waveguide 1/2 in the form of a mode 6. It does not

matter whether the laser beam 5 strikes the diffraction grating 4 from the substrate side or from the measurement substance side. Suitable lasers for use here include, for example, a helium neon laser or a semiconductor laser. The input condition is a resonance condition and is characterized in that the angle of incidence W1 of the laser beam 5 must be selected accordingly, depending on the waveguide configuration, i.e., the effective refractive index of mode 6, to achieve a maximum intensity of mode 6. The angle of incidence W1 of the laser beam 5 is consequently determined by the effective refractory index N of the excited mode 6, which is determined essentially by the refractive indices of the media involved in the waveguide, the refractive index of the measurement substance 3 and the layer thickness of the waveguide film 1. If, due to a change in the refractive index of the measurement substance 3, the effective refractive index N of the mode 6 changes, then the angle of incidence W1 selected originally is no longer optimum, so the intensity of the mode 6 changes. Changes in refractive index of a liquid measurement substance 3 may occur due to a (bio)chemical reaction, for example. However, the measurement substance 3 may also be a solid. Then physical processes such as diffusion processes from foreign substances in thin-layer solids, for example, may be determined if they are associated with changes in refractive index. Both the real component and the imaginary component of the refractive index, which can be interpreted as a complex variable, may change. A change in the imaginary component of the refractive index of measurement substance 3 is associated with a change in the light transmission of measurement substance 3. When there are changes in light transmission, the optical path length plays an important role according to Beer's law, so it is advantageous if the measurement substance 3 can also cover the waveguide film 1 outside of the grating region. The change in effective refractive index of mode 6 can then be measured in two ways.

A detector D1 can measure the change in light intensity of mode 6 when there are small changes in the effective refractive index, and then it is possible to calculate the change in the effective refractive index and therefore the change in the refractive index of the measurement substance 3. This measurement method is suitable for measuring effective refractive index changes which are smaller than the half-width of the resonance input curve. The half-width of the resonance input curve depends on the length L of the diffraction grating because of the uncertainty principle (see K. Tiefenthaler and W. Lukosz, *Optics Letters* 9 (1984), 137-139). At a grating length L = 6 mm and a wavelength of 633 nm, changes in refractive index of the measurement substance 3 on the order of magnitude of 10^{-5} can still be resolved (see dissertation by K. Tiefenthaler, ETH No. 7744).

When there are changes in the effective refractive index amounting to more than the half-width of the resonance input curve, results are recorded by optimizing the light intensity of the mode 6 by readjusting the angle of incidence W1 of the laser beam 5 so that the light intensitv of

mode 6 is always maximized. The change in the effective refractive index of mode 6 can be deduced from the change in the angle of incidence W1. There is also the possibility of selecting the angle of incidence W1 on the basis of calculations, so that a mode 6 of maximum intensity occurs only when the change in refractive index of measurement substance 3 has reached a desired value.

Figure 3 shows an inventive measurement device having a grating output coupler. Waveguide 1/2 and diffraction grating 4 are described in Figure 1. If a mode 6 strikes diffraction grating 4, the laser light is partially or completely output. The output laser beam 7 emerges from the waveguide 1/2 at a certain output angle W2, which is determined by the effective refractive index of mode 6. The generation of mode 6 is not depicted in Figure 3. Mode 6 can be excited, for example, by end face input, prism input, grating input, etc. (see T. Tamir, *Integrated Optics*, chapter 3). A change in the refractive index of the measurement substance 3, which covers at least the grating region, produces a change in the effective refractive index of mode 6 in the grating region, which results in a change in the output angle W2. This change in the output angle W2 can be measured with a diode array D2, for example, or with another position-sensitive detector.

Figure 4 shows a so-called Bragg reflector. The diffraction gratings used for the grating couplers (Figures 2 and 3) may also be used as Bragg reflectors. A mode 6 is reflected on a diffraction grating 4 if the Bragg condition is met, i.e., if the angle of incidence W3 corresponds to the Bragg angle (see W. Lukosz and K. Tiefenthaler, *Optics Letter* 8 (1983), 537-539). With regard to generation of mode 6, the discussion of Figure 3 also applies here. Detectors D3 and D4 measure the intensity of a mode 8 reflected on diffraction grating 4 and/or the intensity of a transmitted mode 9. The Bragg angle is defined by the effective reactive index N of mode 6 in the grating region. If the effective refractive index N of mode 6 changes because of a change in the refractive index of measurement substance 3, then the Bragg condition is disturbed. The intensity of the reflected mode and the intensity of the transmitted mode also change. By measuring the light intensity of the reflected mode 8 and/or the intensity of the transmitted mode 9 with the detectors D3 and/or D4, it is possible to deduce the change in refractive index of the measurement substance 3.

Another measurement option is to select the angle of incidence W3 so that the Bragg condition is not met, but just barely, and therefore there is no reflected mode 8. When the change in refractive index of the measurement substance 3 reaches the desired value, a reflected mode 8 occurs, because then the Bragg condition is met. The change in refractive index of measurement substance 3 can be calculated from the angle of incidence W3 and the intensity of the reflected mode 8 and/or transmitted mode 9.

The mode can be attenuated so much due to light scattering or absorption of the mode on the measurement substance 3 after leaving the grating region that the light intensity can no longer be measured. To minimize or prevent this effect, it is advantageous – as illustrated in Figure 5 – to cover the waveguide film 1 with a protective layer 10 outside of the grating region. This protective layer 10 may be an SiO_2 layer, for example. The layer thickness of the protective layer 10 must be large so that little or no interaction of the mode with the measurement substance 3 is possible outside of the grating region. The protective layer 10 may also be used to prevent any interfering influence of the fastening device used with a cell (not shown in Figure 5) filled with measurement substance 3.

Figure 5 also shows that it is advantageous to for protective layer 10 to increase in the form of a taper (i.e., not in the form of abrupt step) outside of the grating region.

Figures 2 and 4 show detectors for direct measurement of the intensity of modes 6 and/or 8 and 9. Figure 6 shows another detection option; in this case the scattered light 11 generated by mode 6 is captured by fiber optics 12 and sent to a detector D5. The intensity of scattered light 11 is proportional to the intensity of mode 6. There is always scattered light 11 due to unavoidable inhomogeneities in waveguide film 1.

Instead of directly measuring the intensity of the modes 8 and/or 9 with the Bragg reflector (see Figure 4), it is also possible in the same way to measure the intensity of the scattered light of the reflected mode 8 and/or the transmitted mode.

However, as shown in Figure 7, it is also possible for mode 6 to be output first with a second diffraction grating 13, for example, and then the intensity of laser beam 15, which is output at an angle W4, can be measured by a detector D6. This intensity is proportional to the intensity of mode 6. The output mechanism of the second diffraction grating 13 must not be disturbed by the measurement substance 3. For example, this can achieved by having a protective layer 14 separating the waveguide from the measurement substance 3 in the region of the second diffraction grating 13 or having no measurement substance 3 present at all in this grating region (for details on the protective layer, see the discussion of Figure 5). However, the output may also be accomplished via a prism coupler or a taper (see T. Tamir, *Integrated Optics*, chapter 3).

The sensitivity of the integrated optical sensor can be defined as a differential change in the effective refractive index based on a differential change in refractive index of measurement substance 3. Especially high sensitivities are achieved when waveguide film 1 has a much higher refractive index than substrate 2 of measurement substance 3 and when the layer thickness of waveguide film 1 is slightly more than the minimum thickness. A minimum layer thickness of the waveguide film 1 (called the cut-off layer thickness) is required to excite a mode in waveguide film 1 at all (see T. Tamir, *Integrated Optics*, Springer, Berlin, 1979, chapter 2).

To achieve the highest possible sensitivity, it is advisable to select the refractive index of the waveguide film 1 to be at least 1%, preferably more than 10%, greater than that of the substrate 2. However, if changes in refractive index of a measurement substance 3 whose refractive index is greater than that of substrate 2 are measured, then a high refractive index difference between the waveguide film 1 and the substrate 2 is irrelevant for achieving a high sensitivity (see dissertation by K. Tiefenthaler, ETH No. 7744).

The electromagnetic field of a mode as a transversely attenuated wave interacts with the measurement substance 3 and accordingly penetrates into the measurement substance 3 by less than one wavelength, so changes in refractive index can be determined on very small quantities of measurement substance.

Fig. 1

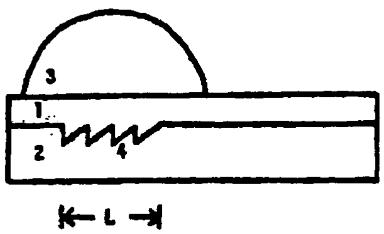


Fig. 4

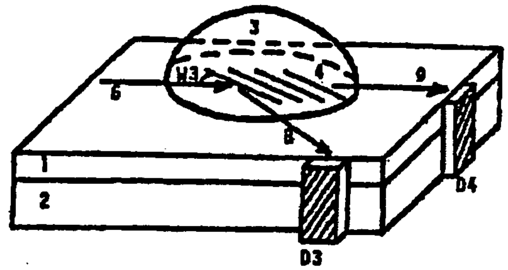


Fig. 2

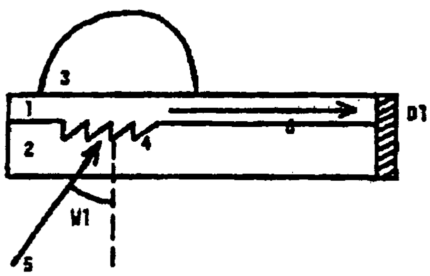


Fig. 5

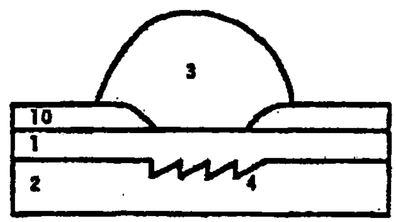


Fig. 3

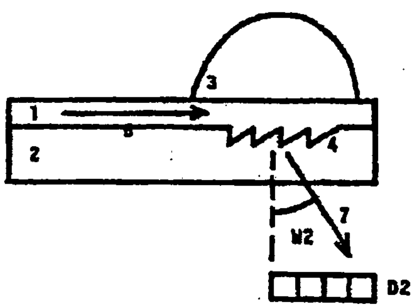


Fig. 6

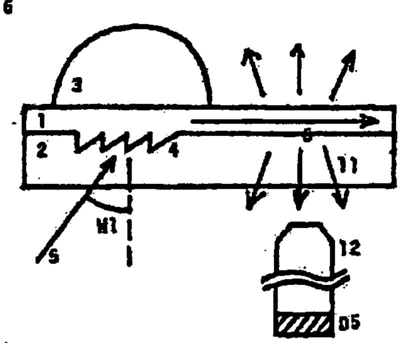


Fig. 7